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Chemical Treatment of Dairy Manure Using Alum, Ferric Chloride and Lime

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Abstract. *Chemical treatment of dairy manure with alum [$Al_2(SO)_4)_3$], ferric chloride [$FeCl_3$] and lime is capable of achieving good solid-liquid separation and concentrating phosphorus [P] in the solid phase. Batch level jar tests conducted using dairy manure containing 0.8 % and 1.6% total solids [TS] indicate that very high removal [>90%] of dissolved reactive P [DRP], total dissolved P [TDP] and total P [TP] can be achieved with chemical addition. In the absence of coagulants, 43% of TP and 30% of TS were removed via gravity settling. Treatment performance varied with chemical type, dosage rate and the initial manure solids concentration. $FeCl_3$ and alum were similar in their efficiency to separate P and solids from the manure slurry. At 8 mM as Al dosage rate for 0.8% TS, alum reduced solution DRP, TDP, and TP levels by 99%, 92%, and 92%, respectively. Lime was less effective than alum/$FeCl_3$ in concentrating TDP and TP with the trend being reversed for TS. At 40 mM as Ca dosage, lime removed 96%, 70% and 69% of DRP, TDP and TP, respectively, for 0.8% TS. Under all treatment conditions, TS and TP removal levels were lower than the extent of removal of other solid and P forms. At the same coagulant dosage, separation of solids and P decreased with an increase in initial manure solid concentration [from 0.8% to 1.6% TS].*

Keywords. alum, $FeCl_3$, lime, phosphorus, solids, coagulation, dairy manure.

I. INTRODUCTION

The most common mode of utilization of animal manure involves land- based disposal. Repeated applications of animal manure [based on nitrogen (N) requirements] have resulted in a build-up of soil phosphorus [P] levels to exceed crop requirements (Sharpley et al., 1999). Due to changes in animal farm dynamics and impending P-based regulations, the available land resources for manure management, particularly near the farms, are going to be seriously stressed (Converse et al., 2000). Therefore, it is imperative to develop practices that will help minimize nutrient loading in areas near the farm [with high P levels] and export the excess nutrients off-farm. The low solids content of animal manure imposes a serious constraint to exporting P to farms that are required to buy fertilizers to meet nutrient requirements (Vanotti and Hunt, 1999). In addition, dilute manures reduce the waste holding times in storage ponds and lagoons. Concentration of organic solids as well as nutrients [mainly P] in a smaller volume will increase manure management options. Physical separation using gravity-based sedimentation basins and mechanical separators offer an economical way to achieve solids-liquids separation (Zhang and Westerman, 1997). These methods are, however, capable of removing only low amounts of solids [5-30%] and P [<10%] (Converse et al., 2000). Therefore, there is clearly a need to investigate the use of alternate manure treatment methods.

Chemical treatment of municipal/industrial wastewater with coagulants [salts of aluminum (Al), iron (Fe), calcium (Ca)] and flocculants [organic polyacrylamide (PAM) polymers] has been shown to be highly successful in removing organic solids [colloidal and dispersed] and P. Soluble P either forms insoluble phosphates [e.g., $AlPO_4(s)$; $Ca_5(PO_4)_3OH(s)$] or adsorbs onto [hydr]oxides of the cationic salts [e.g., Al or Fe (hydr)oxides] (Goldberg and Sposito, 1985). Recent applications of such chemicals to treat animal manure show significant improvement in solids and P removal compared to physical separation methods (Powers et al., 1995; Zhang and Lei, 1998; Vanotti and Hunt, 1999; Sherman et al., 2000; Worley and Das, 2000). Previous studies on chemical treatment have not accounted for all solid and P forms that have potential to impair water quality. For example, most studies did not report the extent of DRP removal. This fraction of P is immediately bio-available and, hence, exerts maximum impact on the environment. Therefore, the major goal of this study is to evaluate the effectiveness of chemical treatment to concentrate P and solids in dairy manure. Specific objectives are to: (i) determine the ability of alum, ferric chloride, and lime to achieve efficient separation of various forms of P [dissolved reactive P (DRP), total dissolved P (TDP), and total P (TP)] and solids [total solids (TS) and total suspended solids (TSS)], and (ii) evaluate their treatment performance as a function of manure solids concentration and chemical dosage.

II. BACKGROUND

Municipal wastewater and water treatment plants use treatment strategies involving coagulation, flocculation and clarification to separate particulates such as clay and silt, natural organic matter, and pathogens from water. Coagulation is a complex process in which chemicals [coagulants] are added to promote the aggregation of suspended particles [colloidal and dispersed] to form larger and heavier solids that settle rapidly (Letterman et al., 1999). Examples of common coagulants include the salts of Al, Fe, and Ca. Flocculation is a physical process that produces interparticle contacts to make particles larger in size and effectively separate the solid phase by sedimentation, sludge blanket clarification and/or filtration. Alum [aluminum sulfate, $Al_2(SO4)_3 \bullet 18H_2O$], ferric chloride [$FeCl_3$], ferric sulfate [$Fe_2(SO_4)_3 \bullet 9H_2O$], polyaluminum chloride, polyaluminum hydroxychlorosulfate and lime [CaO] are among the chemical coagulants used for water and wastewater treatment.

When added to water, alum dissociates into Al^{3+} and SO_4^{2-}. The aluminum ion hydrolyzes to form soluble monomeric [Al^{3+}, $Al(OH)^{2+}$, $Al(OH)_2^+$, $Al(OH)_3^0$, $Al(OH)_4^-$] and polymeric hydroxy species, and amorphous aluminum solid precipitates (Dempsey et al., 1984). The hydrolyzed Al species can interact with organic solids and P in animal manure via precipitation, adsorption and complexation processes. The monomeric species are capable of precipitating P as Al phosphates. Solid amorphous Al precipitates can remove P and solids by adsorption [chemical interaction] and/or physical entrapment. The Fe hydrolysis chemistry is similar to that of Al in terms of (i) the formation of hydrolysis products, (ii) transformations of monomers to polymers, and (iii) the reaction mechanisms between hydrolysis products and P and organic solids. The important Fe hydrolysis products s are: monomeric ions [Fe^{3+}, $Fe(OH)^{2+}$, $Fe(OH)_2^+$, $Fe(OH)_4^-$] and solid precipitate [($Fe(OH)_3(s)$)] (Letterman et al., 1999). The practice of using Ca based compounds for wastewater treatment is not as old and common as Al and Fe salts. Interest in using Ca-rich compounds such as CaO, $Ca(OH)_2$, $CaCO_3$ and industrial by products [fly ash, gypsum] (Moore and Miller, 1994; Stout et al., 1998) for manure treatment is due to their ability to precipitate P as insoluble Ca phosphates. Organic and synthetic polymers [e.g., PAM] are also used as flocculant aids.

Coagulation-flocculation treatment strategies can also be applied to treat animal manures. The purpose of this treatment system is to promote aggregation of the dissolved and suspended manure solids that would be subsequently separated from the suspension by gravity. Nutrients can be separated from the liquid fraction and concentrated in the settled sludge. Zhang and Lei (1998) applied alum, $FeCl_3$ and cationic PAM polymers to treat dairy and swine manures and obtained optimal coagulant dosage and the solids settling rate. The optimal dosage and the corresponding zone settling rate to treat dairy manure [2% TS] are 1000 mg/L and 0.9 cm/min, respectively. Phosphorus removal levels ranged from 53% for treatment with PAM alone to > 95% for combined $FeCl_3$/PAM addition. Sherman et al. (2000) tested alum, $FeCl_3$ and PAM to remove P from dairy wastewater. Treating 1% TS manure with alum [317 mg Al/L], they reported 92% TS recovery and a reduction in P from 143 mg in the original slurry to 4.4 mg. Ferric chloride addition at 376 mg Fe/L recovered 77.8% TS and had effluent P of 16.0 mg. Optimal pH for maximum solids recovery was reported to be 6.41 and 7.58 for alum and $FeCl_3$, respectively.

Moore and Miller (1994) observed a significant decrease in water soluble P from poultry litter treated with inorganic metal salts. Their results were based on incubation studies in which poultry litter was amended with Al, Ca and Fe compounds for 1 week at 25°C. For example, amendment with CaO decreased water soluble P from >2000 mg P/kg to <1 mg P/kg. Jones and Brown (2000) reported 18-35 % TS and 48% TP removal by gravity settling for 2 h from dairy manure containing ≈1.7% initial TS. However, addition of 10 mM Al/L alum significant resulted in a significant increase [93-95%] in P removal.

III. MATERIALS and METHODS

3.1 Site description and Sample information:

This study was conducted using flushed manure collected from a dairy farm near Madison, WI. The facility contains about 1000 cows housed in two free stall barns with mattress and recycled manure solids. The manure slurry from the barn is flushed twice a day to a storage tank and then pumped to pass through a mechanical solid separator. The effluent from the separator is stored in a two-stage lagoon, where some solids settling will occur. The secondary lagoon effluent is pumped for irrigation and/or recycled back to flush the barn. Samples for our study were collected using buckets at the outlet of the primary lagoon. All samples were stored at 4°C to prevent digestion and dissolution of solids and organic nutrients

(Vanotti and Hunt, 1999). Detailed solids and nutrient characterization [electrical conductivity (EC), pH, TS, TSS, TDS, TP, TDP and DRP] data was obtained.

3.2 Bench Scale Jar Tests:

Bench scale tests were conducted using PHIPPS & BIRDS PB-900™ Programmable Jar Tester (Fig. 1). Jar test is the most commonly used procedure to evaluate coagulation process for water/wastewater treatment (Hudson, 1981). The jar tester consists of 6 adjustable paddles for mixing and six rectangular 2-liters jars [21cm x 11.5 cm x 15cm] with an outlet at a height of 4 cm from the bottom for sample collection. A typical jar testing scheme consists of three sequential stages: rapid mixing, slow mixing and sedimentation. The first stage is short with relatively rapid agitation followed immediately by a longer and slow mixing period. Rapid mixing ensures complete coagulant dispersion and also initiates collision of coagulants and primary particles. The slow mixing [2nd stage] process tends to promote the interaction of particles to form dense flocs that can be separated subsequently by settling [3rd stage]. The jar tester was programmed to run sequentially at 100 rpm for 2 min for rapid mixing, followed by slow mixing at 35 rpm for 15 min and finally the flocs were allowed to settle for 30 minutes without any agitation. All chemicals were added to the jars during the rapid mixing stage.

Figure 1. PHIPPS & BIRDS PB-900™ jar tester.

The experiments were conducted at two manure TS levels [0.8 % and 1.6%, wet basis] with the coagulants added at different dosages. Control experiments [no coagulant addition] were also conducted to evaluate solids and P removal in the absence of any chemical addition. All experiments were conducted in duplicates. Initially, wet manure slurry was added to each jar to achieve the target TS level. Depending on the amount of chemicals to be added during rapid mixing, the jars were filled with MILLIQ-grade de-ionized water to bring the final volume to 1.0 L. A stock solution of alum [200 mM as Al] was prepared and a fraction of the stock solution was transferred to the jars to achieve the desired chemical dosages: 1, 2, 4, 8, 12 and 16 mM as Al for both 0.8% and 1.6 % TS. Stock solutions of lime and $FeCl_3$ contained 400 mM as Ca and 200 mM as Fe, respectively. The lime dosages used were 1.25, 5, 10, 20, 40, 60, and 80 mM as Ca and that of ferric chloride were 1, 2, 4, 8, 12, 16 and 20 mM as Fe for both the TS levels.

3.3 Analysis:

Removal of P and organic solids were determined by analyzing the supernatant solutions and settled solids. At the end of the sedimentation period, the height of the settled sediments was recorded. Then, sufficient supernatant solution was collected and stored at 4°C prior to solids and chemical analysis. Solids analyses include measuring TS accumulated at the bottom of the jars, and TSS and TDS levels in the supernatant. After collecting the supernatant by allowing the suspension to drain up to the outlet of the jar, the remaining settled solids were transferred to pre weighed glass beakers and oven dried at 105°C for 48 h to determine the TS content. Percent TS removal was calculated by comparing the dry weight of settled sediments with the summed weight of the initial TS in the manure slurry and the settled chemical precipitates [hydroxides of Al, Fe, and Ca oxide/carbonate]. Due to the difficulty in measuring the exact amount of chemical precipitates in the settled sediments, the following method was used. We assumed that amorphous Al hydroxide [$Al(OH)_3(am)$], and amorphous Fe hydroxide [$Fe(OH)_3(am)$] were formed from the addition of alum and $FeCl_3$, respectively. Assuming complete precipitation of all the Al and Fe that were added, the weight contribution from the chemical precipitates was determined. For lime addition, we took the weight of CaO solid to calculate TS. The following equation was used for total solids analysis:

$$TSremoval(\%) = \frac{SettledSolidsDryWeight(g) - Chemical\ PrecipitateWeight(g)}{TSinitialManure(g)} \qquad (1)$$

Supernatant samples were centrifuged initially at 10,000 rpm on a Sorvall Super T 21 high-speed, bench-top centrifuge for 20 min and then passed through 0.45μm filters. For TDS determination, measured volume of filtrate was transferred to a pre-weighed aluminum crucible and dried for 24 h at 104°C. TSS was calculated from the difference between TS and TDS. The percent TSS removal was calculated by comparing the TSS level in control [no chemical addition] to the TSS concentration in the supernatant after chemical treatment.

Chemical parameters determined for the coagulation experiments include pH, EC, DRP, TDP and TP. These parameters were measured before and after chemical treatment. pH and EC were measured using a Accumet AR-50 pH/conductivity meter. Supernatant samples filtered through 0.45μm were used for DRP and TDP analysis. The filtrate was directly used for DRP measurements using a Lachat Autoanalyzer [Zellweger Analytics, Milwaukee, WI] by following the standard molybdate-based colorimetric methods at a wavelength of 880 nm (Murphy and Riley, 1962). For TDP and TP analysis, the filtrate and unfiltered supernatant samples were digested using a sulfuric-nitric acid digestion procedure (APHA, 1985) prior to analysis. The difference between DRP, TDP and TP concentrations in the control and their corresponding levels after chemical treatment was used to determine % removal.

IV. RESULTS and DISCUSSION

4.1 Manure Characterization:

The mean TS, TSS, and TDS levels are 3.88%, 1.95% and 1.93%, respectively. The N:P ratio in the manure slurry is 7:1. For the manure slurry, DRP concentration is 15.5 mg/L and the particulate phosphorus [PP] concentration, estimated from the difference between TP [255.8 mg/L] and TDP [38.1 mg/L], is 217.7 mg P/L. The PP fraction represents P associated with particles > 0.45 μm and accounts for 85% of the total P.

4.2 Effect of Chemical Treatment on Solids and P Separation:

4.2.1 Alum - P removal

Alum addition significantly reduced the concentration of all the P forms [DRP, TDP and TP] in the supernatant for both 0.8% and 1.6% initial manure TS levels [Figures 2a-2c]. P removal levels shown in Figure 2 are in comparison to the controls [no chemical addition], in which case P separation is mainly achieved by gravity settling of manure solids. Similar removal trends were observed for DRP, TDP and TP as a function of alum dosage; a sharp increase initially at low dosages [up to 4 mM Al] followed by decreasing rates at intermediate dosages before leveling off between 80 and 100 % removal. The recommended alum dosage rate, defined as the dosage above which further chemical addition produced negligible increase in P removal, is 8 mM as Al [216 mg Al/L] for both for 0.8% and 1.6% TS. For 0.8% manure TS, average removal values at the recommended dosage were 99%, 92% and 92 % for DRP, TDP, and TP, respectively. The corresponding values at 1.6% TS were 85%, 86% and 89% for DRP, TDP, and TP, respectively. These results are consistent with those found in the literature. Jones and Brown (2000) treated dairy manure [initial TS of 2.67, 1.89 and 1.32%] with alum and obtained 93-99 % DRP removal at a dosage rate of 10 mM Al/L [reported as optimal rate] using sedimentation periods from 0 to 8 h.

4.2.2 Ferric Chloride - P removal

Figures 3a-3c shows the percent removal of DRP, TDP and TP with $FeCl_3$ addition. For 0.8% TS, the effect of increasing $FeCl_3$ dosage on P removal appears similar to that observed earlier with alum except for the plateau at removal levels exceeding 90%. Since P removal continued to increase with $FeCl_3$ addition, recommended dosage level could not be clearly identified. At 1.6% TS, the sharp increase in P removal at low dosages was not obtained. Complete removal of all P forms from solution can be achieved for the lower initial TS manure at 12 mM as Fe. For 1.6% TS, the maximum DRP and TDP removal was 98% and 93%, respectively, at 20 mM dosage and the corresponding value for TP was 72% at 16 mM. At the higher initial manure solids level, alum appears more effective in concentrating P in the solid phase than $FeCl_3$.

4.2.3 Lime - P removal

Lime appears capable of functioning as an effective coagulant to separate P from the liquid phase of dairy manure [Figures 4a-4c]. For both the TS levels, the recommended dosage rate for DRP is 40 mM as Ca [2240 mg lime/L], with 96% and 92% removal for 0.8% and 1.6% TS, respectively. Even at 20 mM as Ca, half the recommended dosage, significant DRP removal [≈90%] was obtained. For 0.8% TS, maximum TDP and TP removal of ≈90% was obtained at 80 mM as Ca. It also appears that further chemical addition would increase the extent of removal. For 1.6% TS, the recommended dosage for TDP and TP removal is 20 mM as Ca. In comparison to alum and $FeCl_3$, lime is less effective in concentrating TDP and TP in the solid phase. This is especially true for TP; for example, at 1.6% initial TS, 20 mM as Al of alum removed twice as much TP removed by 80 mM as Ca dosage of lime.

Manure solids concentration appears to significantly impact TP and TDP removal by lime addition. TP and TDP removals at 1.6% TS level were significantly lower than those for 0.8% TS. On the other hand, DRP removal is not affected by the increase in manure solids concentration. These differences can be attributed to the predominant mechanism involved in P removal. In the case of DRP, removal could be due to the precipitation of ortho-P as calcium phosphates whereas efficient coagulation/flocculation of suspended solids is necessary to lower TP levels in solution.

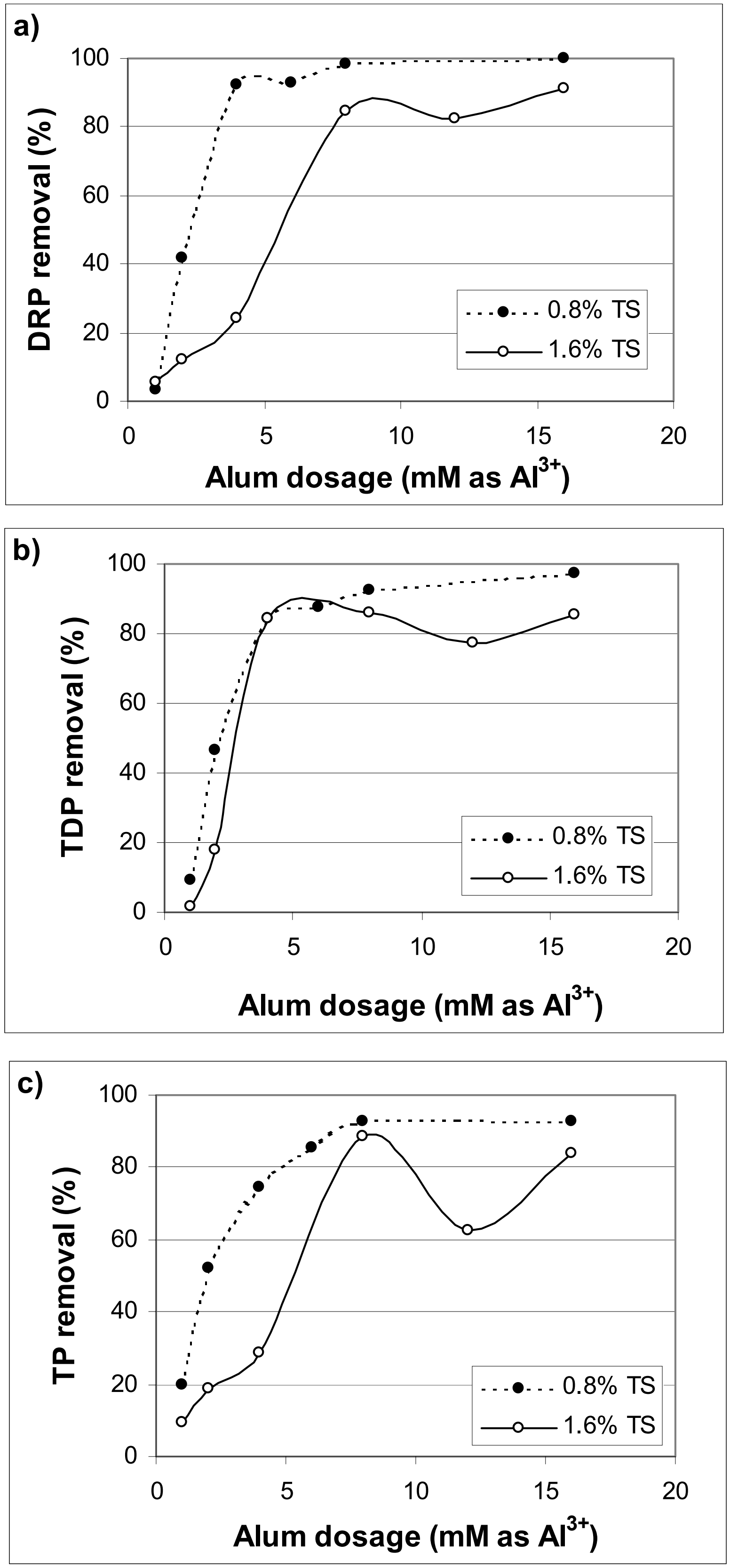

Figure 2. Effect of alum addition on P removal [DRP (a); TDP (b); and TP (c)].

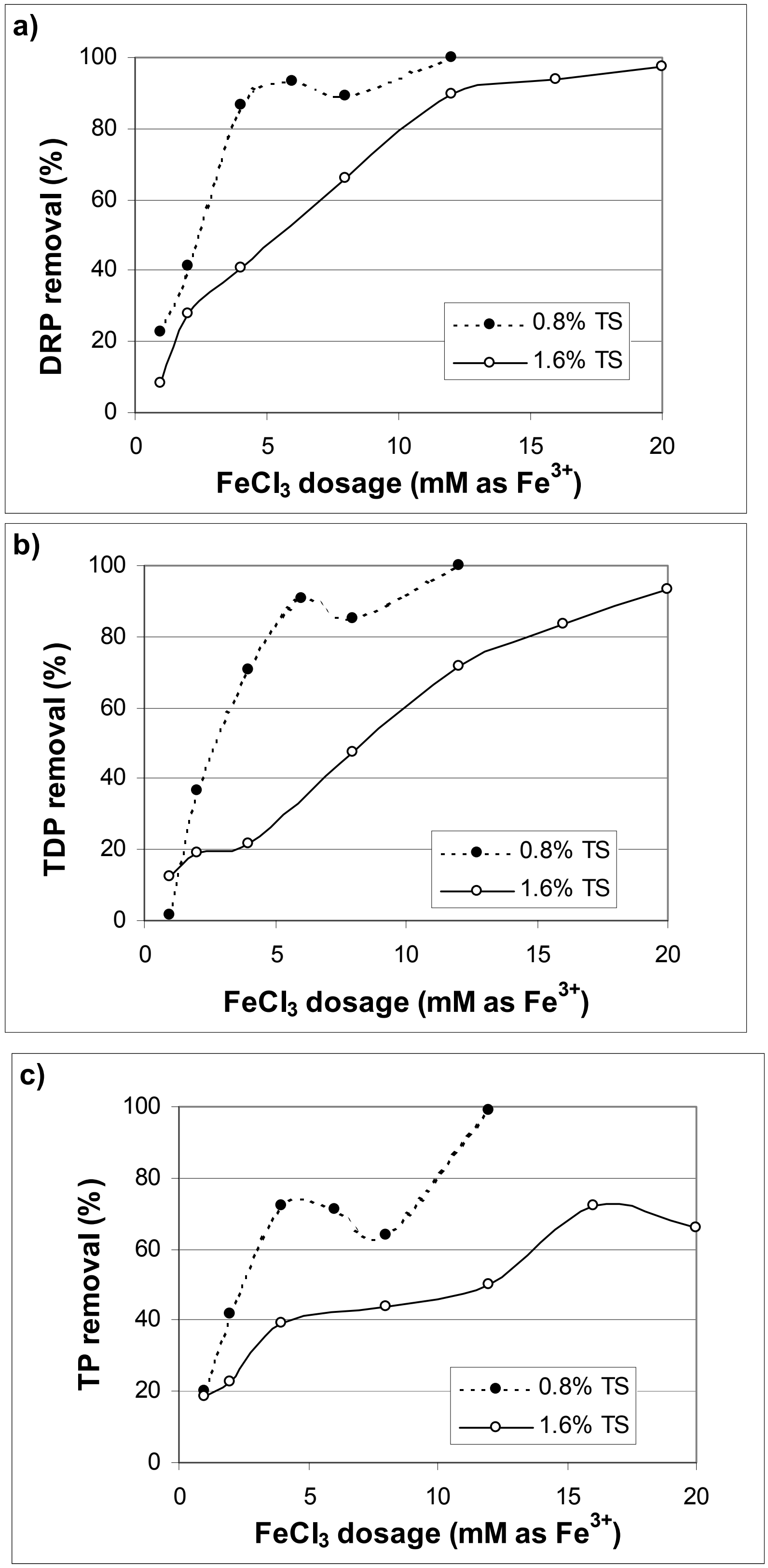

Figure 3. Effect of $FeCl_3$ addition on P removal [DRP (a); TDP (b); and TP (c)].

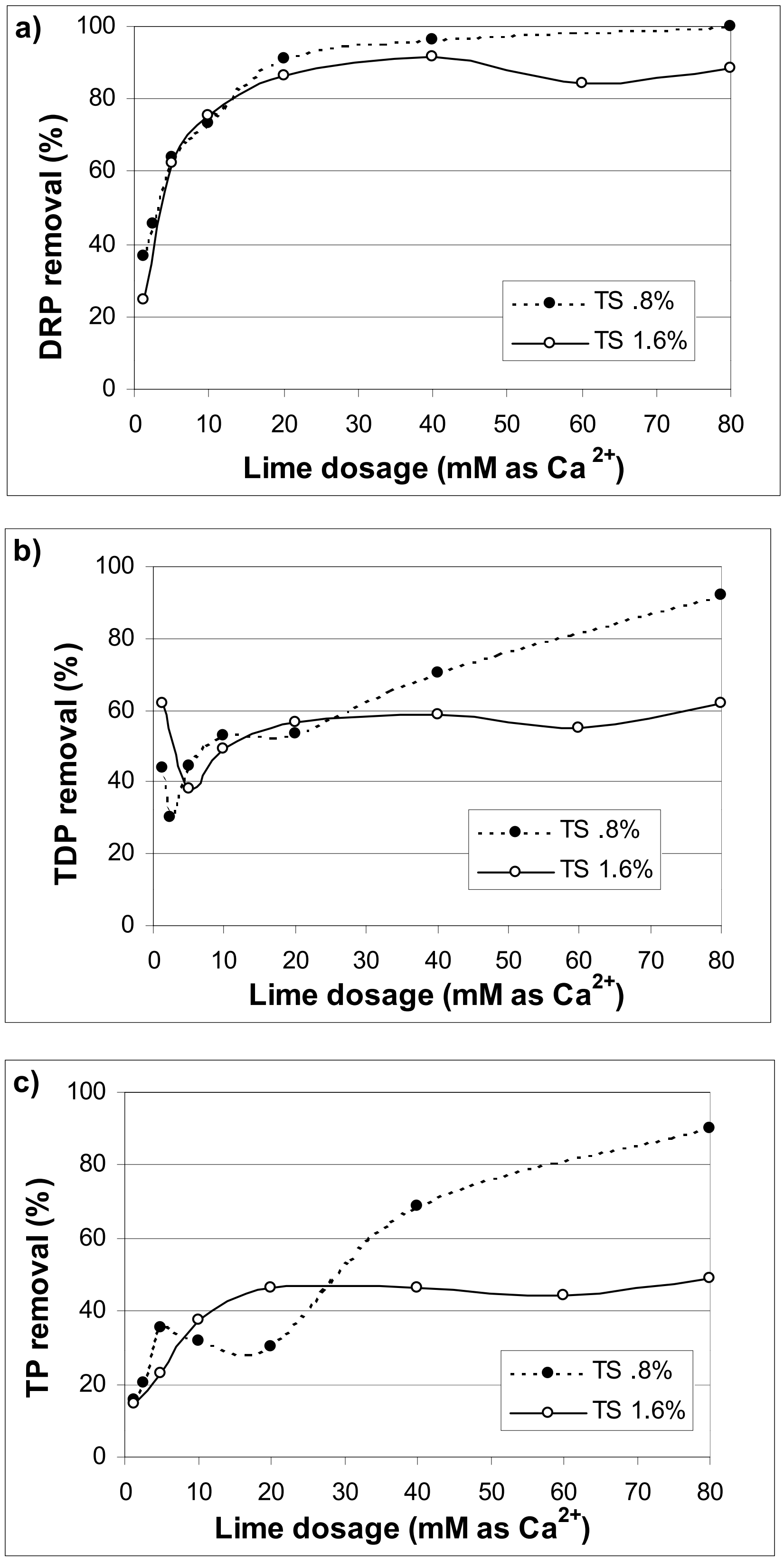

Figure 4. Effect of lime addition on P removal [DRP (a); TDP (b); and TP (c)].

4.2.4 Solids Removal

Solids removal was significantly affected by the addition of chemical coagulants [alum, $FeCl_3$, lime] to the manure slurries. In the absence of coagulants, approximately 15 and 30% of TS was separated from the liquid phase of from the manure slurry for 0.8% and 1.6% initial manure solids, respectively, by simple sedimentation. Chemical addition, depending on the coagulant type, dosage rates and initial manure solid concentrations, further increased the extent of liquid-solid separation. Figures 5a-5c show the TS removal extents for alum, ferric chloride, and lime, which were calculated using levels achieved by gravity settling [i.e., controls] as the baseline.

Percent TS removal increased sharply at low alum and $FeCl_3$ dosages and leveled off at higher dosages. Maximum TS removals achieved with alum were 79% and 65% for 0.8% and 1.6% manure TS, respectively, and similar performance was observed with $FeCl_3$ addition [77% and 70% for 0.8% and 1.6% TS]. Lime was also effective in removing solids [Figure 4], and the recommended dosage is 40 mM as Ca for both the initial manure TS levels. It is interesting to note that at low dosages [< 10 mM as Al or Fe or Ca], lime is more effective in concentrating TS than alum and $FeCl_3$, especially at the higher initial manure solids tested. This trend between coagulants is opposite to that observed for TP, which could be attributed to the differences in removal mechanism for P and solids. TSS removal by alum and $FeCl_3$ was strongly influenced by the initial manure solids level as shown in Tables 1a and 1b.

Table 1. Effect of chemical addition on TSS removal [A- alum; B - $FeCl_3$].

A) Alum:

Alum dosage (mM as Al)	% TSS removal (0.8% initial TS)	% TSS removal (1.6% initial TS)
1	17.3	12.4
2	46.2	24.8
4	71.2	44.5

B) $FeCl_3$:

$FeCl_3$ dosage (mM as Fe)	% TSS removal (0.8% initial TS)	% TSS removal (1.6% initial TS)
1	9.6	13.1
2	40.4	10.3
4	67.3	12.15

V. CONCLUSIONS

This study demonstrates that coagulants [alum, $FeCl_3$ and lime] are capable of concentrating considerable amount of P [different forms, such as DRP, TDP and TP] and solids [TS and TSS] in the solid phase. Alum and $FeCl_3$ appear similar in their ability to remove P and solids at the lower initial manure solids whereas alum was more effective at 1.6% initial TS. Higher dosages of lime are required to achieve similar TDP and TP removals as alum/$FeCl_3$ and this trend between chemicals is reversed for TS. All three coagulants are highly effective in concentrating DRP. For all treatment conditions, solids and P removal was higher for the lower TS level (0.8%) as compared to 1.6% TS. Within each TS level, removal of different P forms followed this order: DRP > TDP > TP.

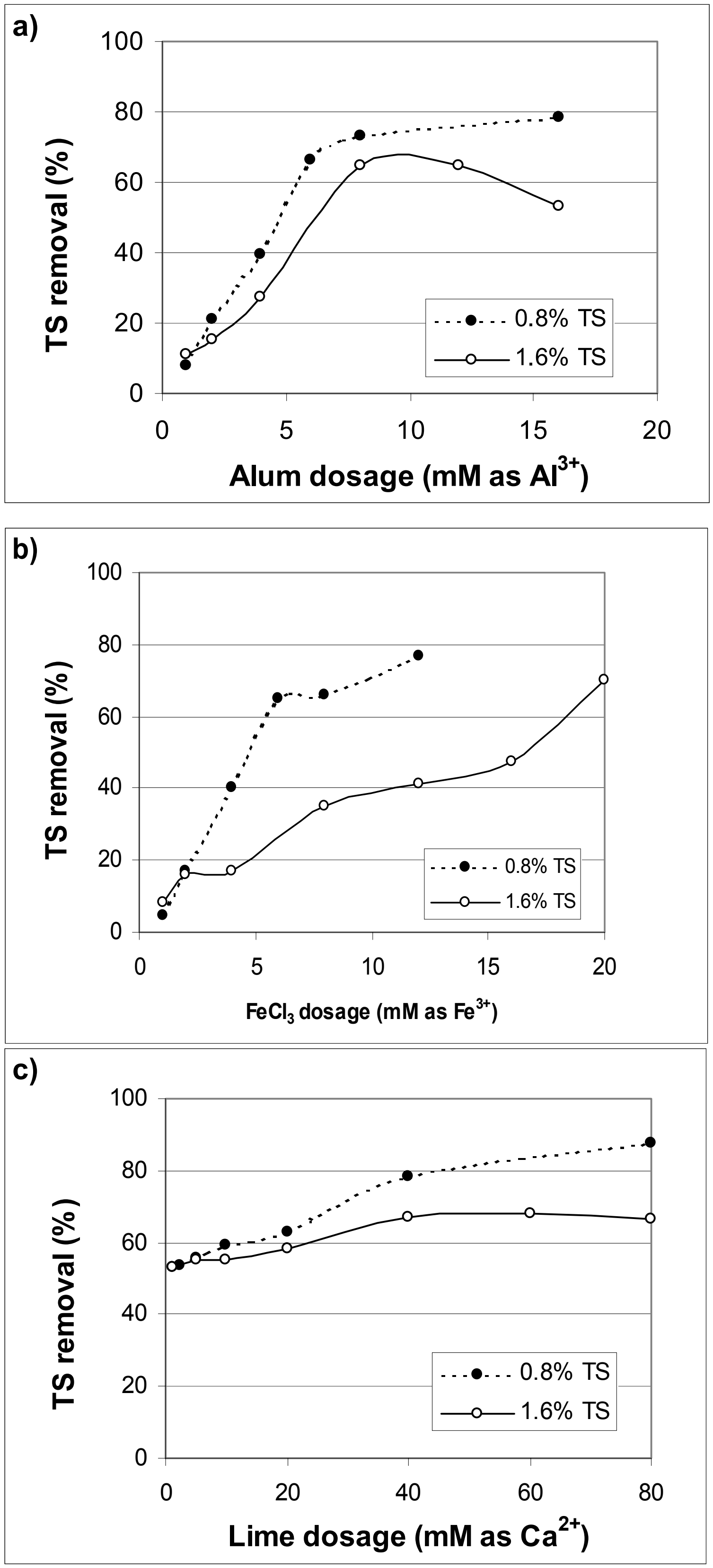

Figure 5. Effect of chemical addition on TS removal [alum (a); $FeCl_3$ (b); and lime (c)].

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